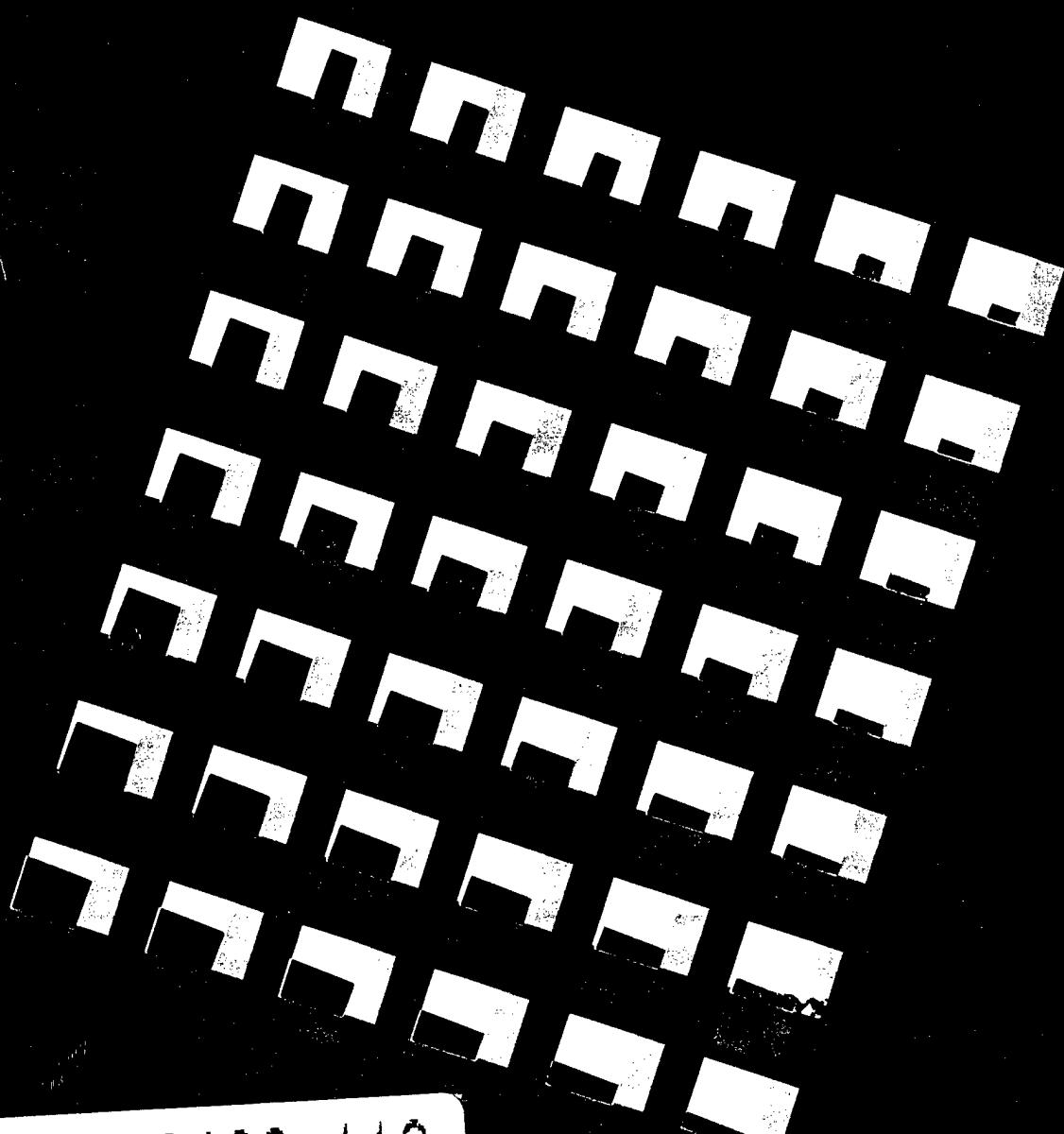


TNO report
PML 1996-A58

The construction of the friability test at TNO-PML

TNO Prins Maurits Laboratory



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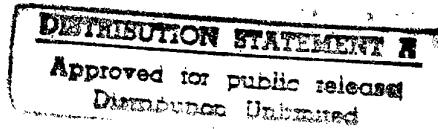
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Managementuitreksel

Titel : The construction of the friability test at TNO-PML
Auteur(s) : Ir. J.H.G. Scholtes en Dr. B.J. van der Meer
Datum : november 1997
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Rapportnr. : PML 1996-A58

Aanleiding van het onderzoek

Als onderdeel van de MOU met Frankrijk is in samenwerking met SNPE de friability test gebouwd. Deze test was niet aanwezig op het TNO Prins Maurits Laboratorium (TNO-PML) en was wel gewenst, aangezien met deze test inzicht verkregen kan worden in de schokgevoeligheid van explosieven. SNPE voert deze test al een aantal jaren uit en met behulp van die kennis kon de test nu worden gerealiseerd.

Doel van het onderzoek

Doel van het onderzoek is het beschikbaar krijgen van de friability test.

Korte omschrijving van het onderzoek

De friability test is gebouwd en getest in samenwerking met SNPE, Frankrijk. Met deze opstelling kunnen thans 'samples' worden getest op de gevoeligheid voor 'impact' op hun brandeigenschappen.

Het apparaat werkt naar tevredenheid.

Conclusies en aanbevelingen

De friability test is nu op het TNO-PML beschikbaar en operationeel. Een klein aantal verbeteringen om de uitvoering te vereenvoudigen is voorgesteld.

Met een negental kruiten zijn experimenten uitgevoerd. De kruiten bleken slecht bestand tegen 'impact', daar de kruiten geheel verpulverden. Twee van verpulverde kruiten zijn vervolgens beproefd in de 'closed vessel'-test en bleken, desondanks, geen ontoelaatbare brandeigenschappen te bezitten. In verder onderzoek zal hieraan aandacht worden besteed.

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1 Introduction

Recently a co-operation between the French and the Dutch research laboratories (SNPE and TNO Prins Maurits Laboratory, respectively) has been established within in a Memorandum Of Understanding. Part of this agreement deals with the construction and testing of the friability test.

The friability test is used to determine the effect of impact on an EIDS candidate [1]. The test is split into two phases. In the first phase, a test sample of 9.0 grams has to be projected against a steel plate at a velocity of about 150 m/s. In the second phase, this impacted sample is tested in a manometric bomb (a closed vessel) to determine the average maximum pressure rise rate, $(dp/dt)_{max}$, of three tests, which is not allowed to be more than 15.2 ± 0.2 MPa/ms.

In Chapter 3 a description of the experimental set-up is given. The chapter is divided into two parts; a description of the gas gun set-up and the manometric bomb. In the following chapters the experimental execution and an evaluation of the first test results are given. The last chapter contains the conclusions.

2 The experimental set-up of the friability test

2.1 Introduction

Figure A.1 shows a construction drawing of the gas gun test set-up placed in an explosion-safe test facility. The gas tank (Figure A.1, part number 2) and the launching tube, together with the two air valves, form the gas gun. The gas tank is filled from the control room by opening the electromagnetic and controlled air valve (part number 1). The air is supplied by a standard buffer air tank of 50 litres and 20 MPa near the control room. After filling, the first air valve is closed and a test sample of 9.0 ± 0.1 grams can be launched by opening a second valve (part number 3). Before the sample enters the recovery box (6), its speed is measured by means of two optical barriers (5). At the end of the recovery box, the sample is stopped by a steel impact plate. After the impact test the recovery box is opened and the sample-fragments can be recovered. If the weight of the fragments is at least 8.8 grams, the fragments are burnt in a modified closed vessel test. During burning, the pressure is recorded as a function of time. From these data, the maximum pressure increase corresponding with an impact of 150 m/s, is obtained. Standard, the test is carried out in triplicate. If the average (of three measurements) maximum pressure derivative is greater than 15 MPa/ms, the test result is positive and the substance is not an EIDS candidate. In the following paragraphs a description of the construction of the test set-up is given. At the end of this chapter a more detailed description of the electrical equipment is given.

2.2 A detailed description of the launching device test set-up

2.2.1 The gas gun in the test facility

The gas gun is put together from two magnetic air valves, a gas tank and a launching tube. Figure A.2 shows a construction drawing of the gas tank. Compared to the gas tank of the SNPE, the tank is modified to decrease the length of the total test set-up. The length of the gas tank is decreased to 500 mm; a quarter of its original length. On the other hand, the inner diameter is increased to 125 mm. With these modifications the original volume is maintained and the functioning of the gas gun will not be changed compared to the French construction. A pressure transducer is mounted onto the gas tank to measure the pressure during the filling process. On the left side of Figure A.1, the gas tank is connected to a standard 50 litre, 20 MPa buffer air tank through an air valve. The valve is remote controlled from the control room. On the left side, the tank is connected to the launching tube through a special fast-functioning valve (part number 3, Annex A). This second valve is also remote controlled but has a much shorter opening time

than the first valve. This way the airflow is not influenced by the functioning of the valve. In Figure A.3 a construction drawing of the launching tube with a length of 1500 mm is shown. The inner diameter is 18.35 ± 0.05 mm. The inner surface is honed to a surface roughness of 0.4 micron to decrease the friction between the sample and the tube during launching. With the length of 1.5 m and a gas tank pressure of 1.2 MPa, a theoretical maximum velocity of 230 m/s can be reached with a standard sabot and test sample mass, depending on the friction coefficient.

The total length of the modified test set-up is about 5.5 metres. The internal length of the test facility (bunker) is about 4 metres. Therefore the gas gun is mounted to a frame outside the bunker (see Figure A.1). The launching tube enters the bunker through a hole of 100 mm. A construction drawing of the frame is given in Figure A.4. The gas gun is protected by a PVC cylinder mounted to the steel frame (Photo B.1, Annex B) and closed at one side.

2.2.2 The optical barriers

Before the test sample enters the PMMA recovery box (part number 6, Figure A.1 and Photo B.2, Annex B) the velocity of the sample is determined by measuring the time interval for the passage between two optical barriers (part number 5 in Figure A.1). Figure A.5 shows a magnification of the optical barriers mounted to a frame. The frame is mounted to the concrete wall. This frame can be turned to allow access to the recovery box. The two optical barriers have an internal dimension of 80 x 120 mm. Both barriers produce a short pulse at the moment a projectile passes the barrier. By measuring the time interval between the pulses with a counter, the velocity of the sample can be calculated, knowing the exact distance between the two sensors. More details on the reproducibility of the velocity measurement are given in Paragraph 2.4.1.

2.2.3 The recovery box and the impact plate

In Figure A.6, a more detailed construction drawing of the PMMA recovery box is shown. The box is mounted on a removable frame (see Photo B.2, Annex B).

During testing, the sample enters the box through a PMMA circular plate with a hole of 100 mm in the centre of the plate. The plate is removable to simplify the collection of the sample-fragments after testing. The sample is stopped by a steel impact plate with a diameter of 650 mm and a thickness of 20 mm at the end of the recovery box. The impact plate is mounted to the concrete bunker wall. During testing, the recovery box is clamped to the impact plate. In this way, the recovery box is closed at both ends during testing to avoid loss of fragments of the test item.

2.3 The manometric bomb

2.3.1 Introduction

With the closed vessel it is possible to assess the burning characteristics of a propellant or high explosive. Figures A.7 and A.8 show the construction drawings of the closed vessel. The standard closed vessel has an internal volume of 700 cc. For the friability sample testing the vessel is modified. After the sample is placed in the vessel, the vessel is closed and the ignition plug can be mounted into the vessel. After firing, the internal pressure is recorded as a function of the time. This enables the calculation of the maximum pressure derivative $(dp/dt)_{max}$ corresponding with an impact speed of 150 m/s. Standard, three tests are carried out for each type of material. Because the closed vessel test is a standard test and only some minor modifications are made for friability testing, a short description of the modification and a description of this test is given in the following paragraphs. In the work instruction of the closed vessel test [2], a more detailed description is given.

2.3.2 The modification of the closed vessel test

In Figure A.8, a construction drawing of the centre part of the closed vessel is given. In the lower part of this drawing the metal jacket is shown, which is the modified part of the vessel. In the upper part, the original chamber together with the jacket is shown. The volume of the modified closed vessel should be such that a 9.0 gram 'reference gun propellant' (delivered by SNPE) shows a maximum pressure of 80 ± 2 MPa and a pressure derivative of 15.2 ± 0.2 MPa/ms. This can be performed by gradually changing the volume of the closed vessel. However, neither calibration requirement (peak pressure and $(dp/dt)_{max}$) could be fulfilled as there is only one variable available that can be changed (see Paragraph 3.3).

2.4 The gas gun test set-up

2.4.1 The velocity measurement

The velocity of the sample is measured before it enters the PMMA recovery box. The velocity is obtained by dividing the distance and the time interval needed for the projectile to travel this distance. In Figure A.1 (part number 5), a drawing is shown of the optical barrier (STM RLS 128) used in the gas gun test set-up to determine the sample velocity. The exact distance of the optical barriers was 300.8 ± 0.2 mm. During a velocity measurement the sample passes the optical barriers, which produce a short electrical pulse. Measuring the time interval between the pulses using a chronometer/counter (time-resolution of 1 ns), the velocity of the sample can be obtained.

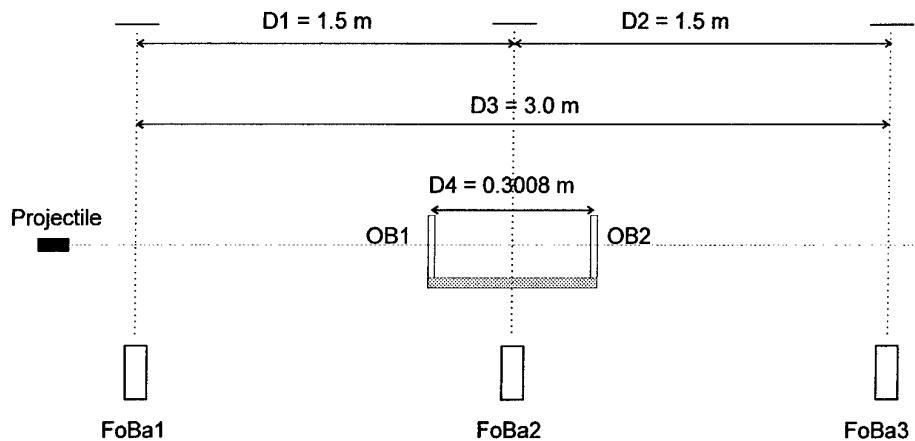


Figure 1: Test set-up for velocity measurement comparison.

According to the specifications of the supplier of the optical barrier, the devices should have a switching delay of $t < 10 \mu\text{s}$ for an object with a diameter greater than 5 mm and a speed in the order of 100 m/s. To ensure the optical barrier works in the proper manner, it was compared with a calibrated high-speed optical barrier used at the TNO-PML's Ballistic Laboratory. In Figure 1, the test set-up for the comparison experiment is shown. D1, D2, D3 and D4 are the space intervals between the different optical barriers. D4 is the space interval of the optical barriers used for the friability test, and D1 to D3 the intervals between the high-speed photo-barriers. A projectile with a diameter of 7 mm was launched from the left travelling to the right. During its passage it is detected by FoBa1, OB1, FoBa2, OB2 and FoBa3, respectively, resulting in five short pulses at different moments in time. In Table 1, an overview of the measured time intervals and the corresponding velocities of the projectile for the different space intervals is given. The time intervals T1 to T4 and velocities V1 to V4 correspond to the space intervals D1 to D4, respectively. The deviation and relative deviation in speed between the optical barrier used in the friability test and the large photo barrier are given in columns 9 and 10. The relative deviation in the velocity is smaller than 0.4% at high velocities ($>400 \text{ m/s}$) and decreases to a value of less than 0.05% for low velocities ($<400 \text{ m/s}$).

Table 1: Results of velocity measurement comparison experiments.

T1 (ms)	V1 (m/s)	T2 (ms)	V2 (m/s)	T3 (ms)	V3 (m/s)	T4 (ms)	V4 (m/s)	d(V3-V4)	rel. error (%)
2.44	614.45	2.50	599.88	4.94	607.08	0.496	606.80	0.275	0.045
2.81	532.92	2.88	521.05	5.69	526.92	0.537	524.91	2.004	0.380
2.84	528.862	2.90	516.67	5.74	522.69	0.577	520.87	1.825	0.349
3.01	498.52	3.08	487.36	6.09	492.87	0.611	492.42	0.444	0.090
3.21	467.76	3.28	456.97	6.49	462.30	0.652	461.33	0.971	0.210
3.31	452.67	3.39	442.89	6.70	447.73	0.672	447.67	0.059	0.013
3.51	426.82	3.59	417.68	7.11	422.20	0.713	422.00	0.197	0.047
4.17	359.91	4.26	352.14	8.43	355.98	0.845	356.14	-0.161	-0.045
4.54	330.65	4.63	324.30	9.16	327.44	0.919	327.38	0.058	0.017
4.67	321.38	4.76	315.36	9.43	318.29	0.945	318.29	0.002	0.0006

2.4.2 The temperature controller

According to the UN manual's friability test description, the test sample should be brought to and kept at a temperature of 20 °C. To preserve this prescribed temperature, a double isolated 300 W heater is mounted to the launching tube. The temperature of the heater is controlled by a Eurotherm 810 temperature-controller. A thermocouple is mounted to the launching tube for a feedback of the temperature. The controller is equipped with a digital indicator, so the temperature of the sample and the tube can be verified up to the moment of firing.

2.4.3 Gas tank filling

Before the test sample can be fired, the gas tank must be filled with air provided by a 50 litre, 20 MPa buffer tank. The buffer tank is equipped with a pressure-reducing valve which reduces the high pressure of 20 MPa down to about 1.5 MPa. To obtain a reproducible sample velocity, a constant gas pressure before firing is essential. To archive this constant pressure in the air tank, the signal of a pressure transducer, mounted to the gas tank, is led to digital controller/indicator in the control room. To fill the gas tank, the pressure controller is pre-set to the required value and the air valve is opened. The valve is automatically closed at the moment the pre-set pressure is reached.

To launch the sample, the second valve must be opened. To ensure the airflow during opening is not influenced by the valve, a special valve with a short opening time is used. This valve is also remote-controlled from the control room and can be activated by pressing the fire button.

3 The experiment

3.1 The gas gun experiment

Before the test sample is brought into the muzzle of the launching tube, the temperature of the tube must be checked not to exceed the value of 22 °C. The pressure of the gas tank should be around normal air pressure of 0.1 MPa. If both values are checked, the 9.0 ± 0.1 g sample together with a special sabot of 1 g (plastic wad) are brought into the muzzle to a prescribed distance with a stick. The bunker door is then closed. The pressure controlling device is set to its prescribed value and the valve is opened by pushing the control button. After reaching the pre-set value, the airflow is stopped. Depending on the weather and temperature conditions, the pressure will decrease or increase slowly. If the pressure increases, the pre-set value must be set below 1.2 MPa; if the pressure decreases, another push on the fill button will bring the pressure up above 1.2 MPa.

Before pushing the fire button, the temperature controller must be checked, indicating a tube temperature of 20 ± 2 °C. Also the time interval chronometer/counter, connected to the optical barriers should be on standby. After pushing the fire button, the pressure will be released and the sample is accelerated. During its flight, the samples speed is determined by the optical barriers and the sample will be stopped by the impact plate. Before entering the bunker after a shot, the pressure of the gas tank should be checked to have a value of 0.1 MPa, for safety reasons. The bunker door is opened, the optical barrier is turned away, so that the top plate of the recovery box can be removed. The sample fragments are gathered to be burned in the modified closed vessel tests.

3.2 The closed vessel experiment

If the mass of the recovered sample fragments, after a gas gun experiment, is at least 8.8 g, the sample is suitable for a closed vessel testing. First, the closed vessel has to be prepared for testing. The vessel, the ignition plug and some other small parts are cleaned with alcohol. A piezoelectric pressure transducer (type Kistler 6211) with a range of 0-750 MPa is mounted into the vessel wall and the modification jacket is positioned inside the chamber. The vessel is preconditioned to a temperature of 21 ± 2 °C. The sample is also preconditioned to the same temperature. Thereafter the sample is placed in the vessel chamber.

After the chamber is closed the ignition plug together with the igniter is mounted into the vessel wall. The pressure release plug is closed and the vessel is ready for testing. After all safety precautions have been taken and the data acquisition sys-

tem is on standby, the igniter is fired. The igniter will ignite the test sample and the pressure is recorded as a function of time. Two minutes after the experiment, the pressure can be released by opening the plug.

After smoothing the raw pressure data, the time-derivative of the pressure can be calculated. From this, the maximum pressure rise can be obtained corresponding to a impact velocity of 150 m/s. For each type of material, three tests are carried out. If the average maximum pressure rise is greater than 15 MPa/ms, the substance tested is not an EIDS candidate and is noted as positive.

3.3 Calibration of closed vessel

The closed vessel is calibrated using the reference propellant GBPa 125, obtained from SNPE. With this reference propellant, the volume of the closed vessel should be adjusted such that the combustion of 9 grams GBPa 125 results in the maximum pressure of 80 ± 2 MPa and the maximum pressure time derivative of 15.2 ± 0.2 MPa/ms. It should be noted that with only one parameter (the vessel volume), *both* the maximum pressure and the maximum pressure rise rate should be adjusted.

The first step was to achieve the maximum pressure of 80 MPa by changing the vessel volume to about 100 cc.

The second step was to calculate the maximum pressure rise rate (dp/dt). However, due to noise superposed on the slope of the pressure curve, dp/dt changes drastically and $(dp/dt)_{max}$ cannot be easily derived directly from the experimental curve. A Fast Fourier Transform and a polynomial curve fitting technique was used to filter the frequencies associated with the noise from the pressure signal. Subsequently, the derivative of the filtered signal was calculated to obtain $(dp/dt)_{max}$. However, depending on the mathematical technique or the portion of the pressure curve used, the $(dp/dt)_{max}$ was 12.3 ± 0.2 MPa/ms, where the error of 0.2 MPa was solely due to the fitting procedure. It is therefore concluded that the 0.2 MPa requirement is too severe, at least for the obtained pressure signal.

The reproducibility of the maximum peak pressure in the experiments is better than 2%, which is within the required 3%.

The experiments with the propellants were performed with the test volume, resulting in the above-mentioned peak pressure and pressure rise rate.

4 First results

In Tables 2 en 3, an overview of the first test result with energetic materials is given. In the first row, the depth of the projectile in the launching tube is given. In rows 2 and 3, the masses of the projectiles before and after the gas gun experiment are given. The time difference of the two trigger pulses of the optical sensors is given in row 4, and the calculated velocity is given in row 5. A closed vessel test is performed for only two samples. For these experiments the maximum pressure and maximum derivative of the pressure are given in rows number 6 and 7.

Table 2: Friability test results of PBX-HMX HU 28.

	FT 9501	FT 9502	FT 9503	FT 9504	FT 9505
Depth in launching tube (cm)	75	35	25	85	55
Mass before (g)	9.0	9.0	9.1	9.1	9.1
Mass after (g)	7.7	8.8	8.8	8.1	8.2
Time (ms)	2.119	2.890	3.205	1.994	2.497
Velocity (m/s)	141.9	104.0	93.8	150.7	120.4
Max. pressure (MPa)	-	71.9	-	-	-
(dp/dt) _{max} (MPa/ms)		7.5	-	-	-

Table 3: Friability test results of a standard rocket propellant.

	FT 9506	FT 9507	FT 9508	FT 9509
Depth in launching tube (cm)	35	55	85	- ¹
Mass before (g)	9.1	9.1	9.1	9.1
Mass after (g)	9.0 ²	9.1	8.7	9.0
Time (ms)	2.970	2.3897	2.037	2.2306
Velocity (m/s)	101.2	125.8	147.6	136.2
Max. pressure (MPa)	-	-	-	58.5
(dp/dt) _{max} (MPa/ms)	-	-	-	6.4

¹ Probably, the depth the sample was put into the tube was 70 or 75 cm.

² One fragment of mass 1.3 gram was retrieved from the floor of the bunker.

Both the PBX-HMX and the standard rocket propellant broke into numerous pieces (almost a powder) in the friability test (inducing a large burning surface) though the velocity of the grain was than the required 150 m/s. Nevertheless of the increased burning surface, the peak pressure and the pressure rise rate remains rather low, as can be seen from Table 2 and 3. If the grains would have survived the test, the value of both parameters would have probably been smaller than the values they are now. However, if the grains would have been subjected to the required velocity of 150 m/s, larger values for the pressure rise rate and the peak pressure would probably have been measured.

5 Conclusions

The friability test set-up has been realised and works satisfactorily. Nine experiments with explosives, a standard rocket propellant and a PBX-HMX, were performed. Both explosives pulverised to a large extent after impact, but showed a moderate burning behaviour in the closed vessel test.

One of the requirements for the calibration of the closed vessel test using the reference propellant could not be fulfilled, as only one parameter is available for adjustment. In the described tests the closed vessel met the requirement for the peak pressure but not the pressure rise rate.

6 References

- [1] 'Recommendations on the transport of dangerous goods'; test and criteria, second edition, st/sg/ac.10/11 rev.1. United Nations, New York 1990.
- [2] Brand, J. van de,
'Uitvoeren van tests met de closed vessel',
internal report, work instruction BR-R52 CLV/01.

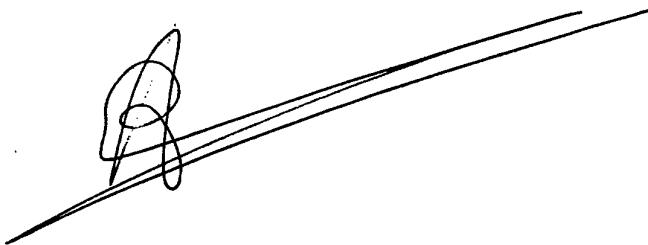
7 Authentication



Dr. B.J. van der Meer
Research Co-ordinator/Author

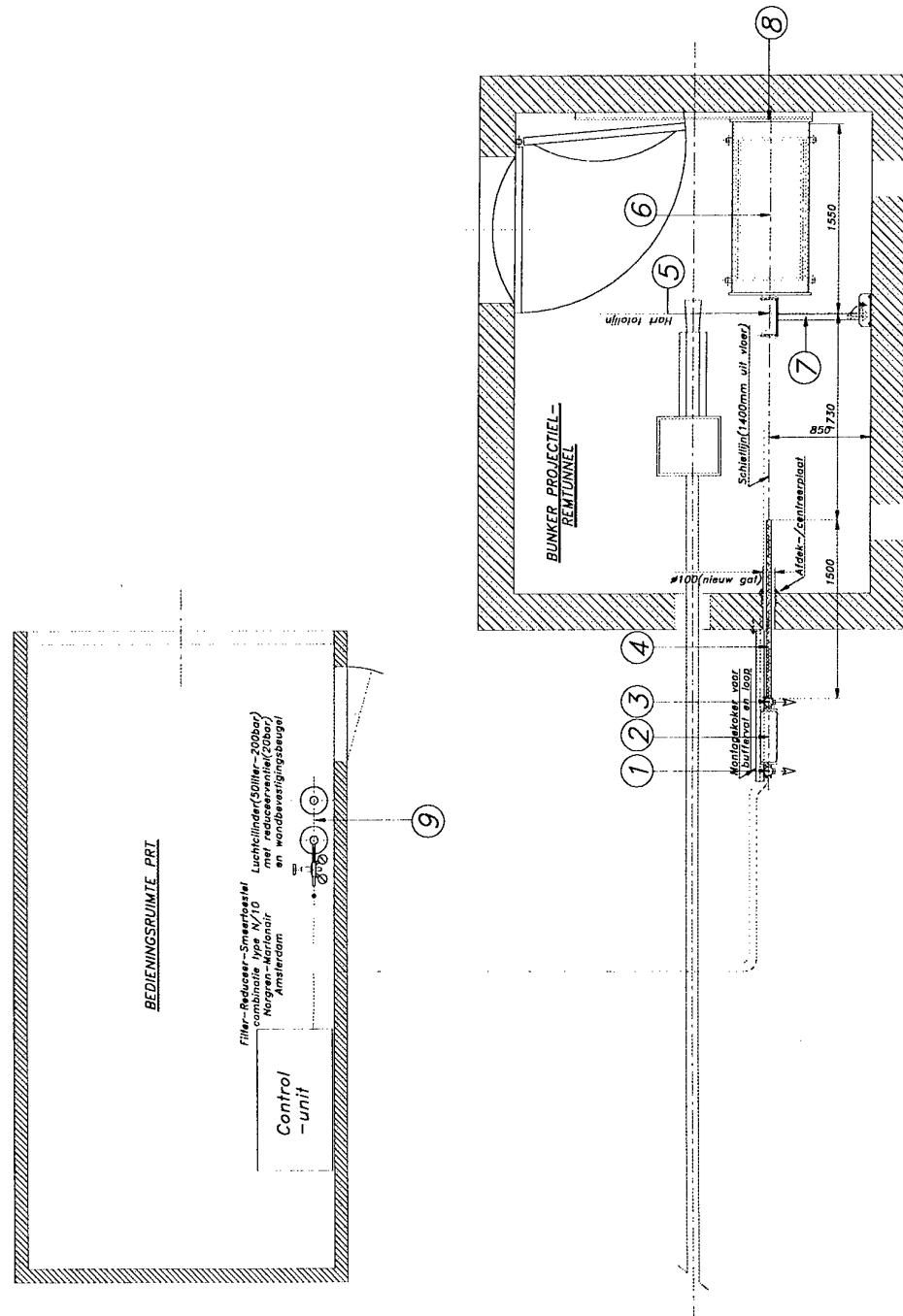


J.H.G. Scholtes
Author



Dr. A.C. van der Steen
Group Leader

Annex A Construction drawings



*Figure A.1: Construction drawing of the gas gun test set-up for the friability test.
1, 3 = Air valves; 2 = gas tank; 4 = launching tube; 5 = two optical barriers;
6 = recovery box; 7 = mounting support for two optical barriers; 8 = concrete
wall; 9 = cylinders for air supply to gas tank.*

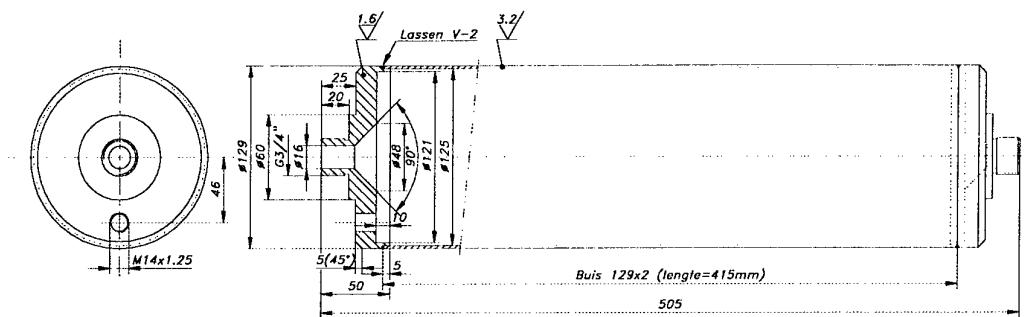


Figure A.2: Gas tank.

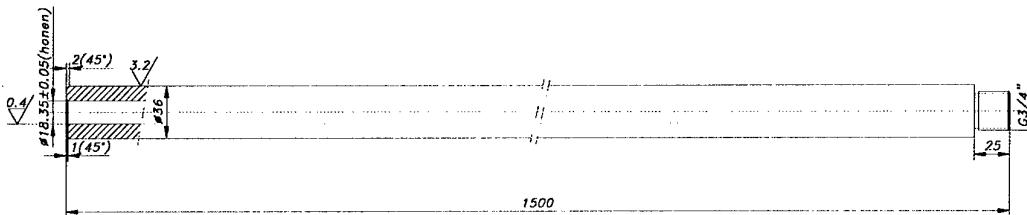


Figure A.3: Launching tube.

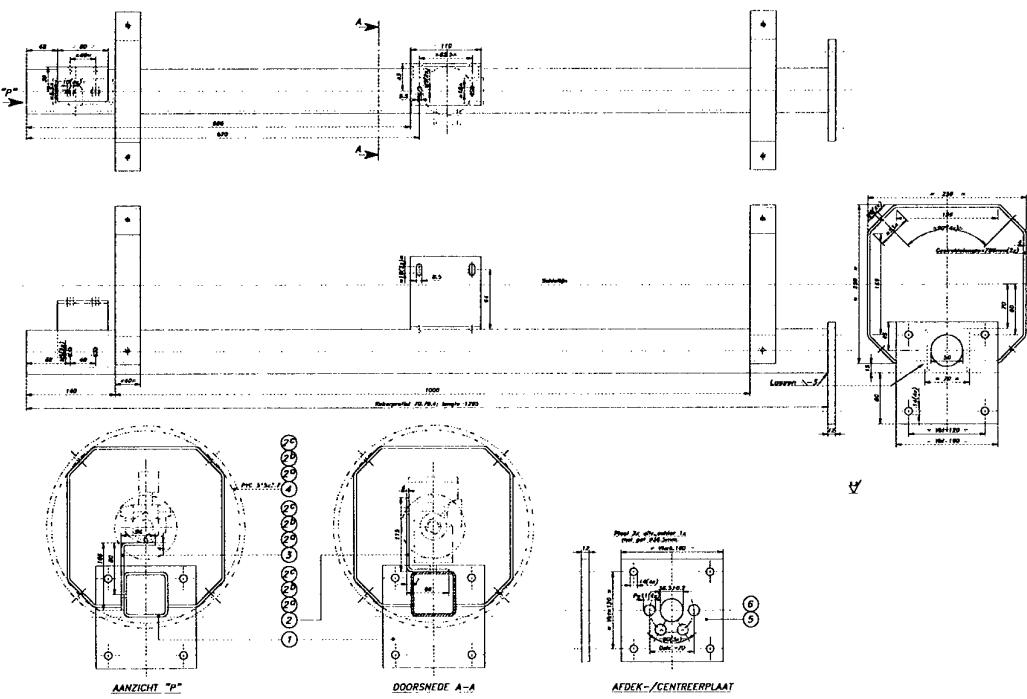


Figure A.4: Suspension of launching tube.

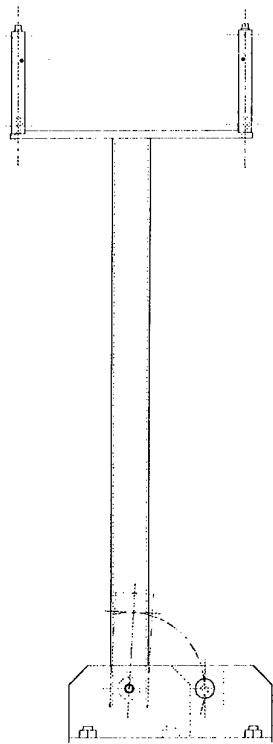


Figure A.5: Rotatable mounting support and optical barriers.

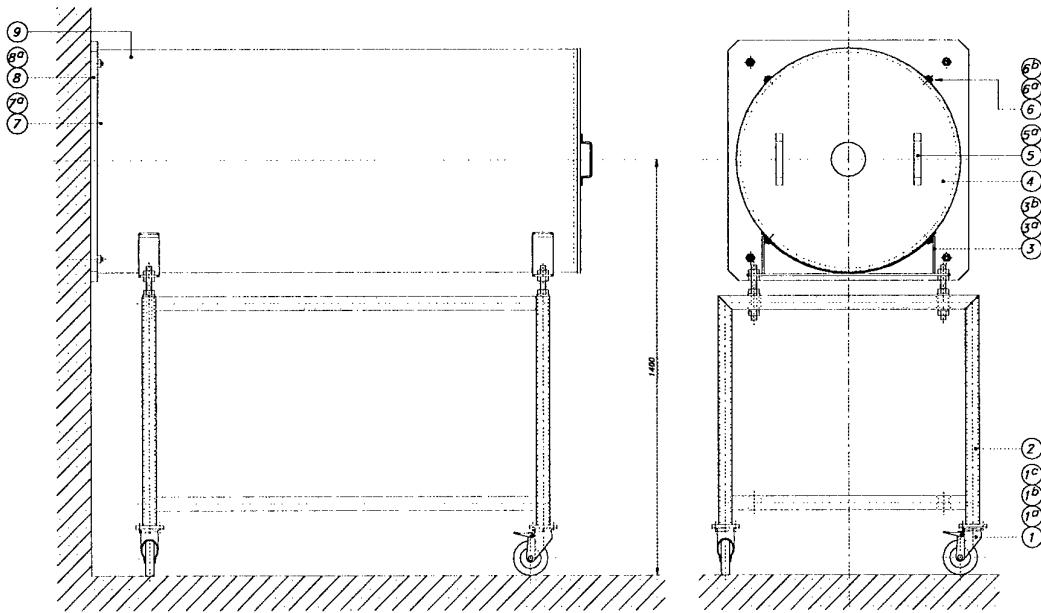


Figure A.6: Recovery box.

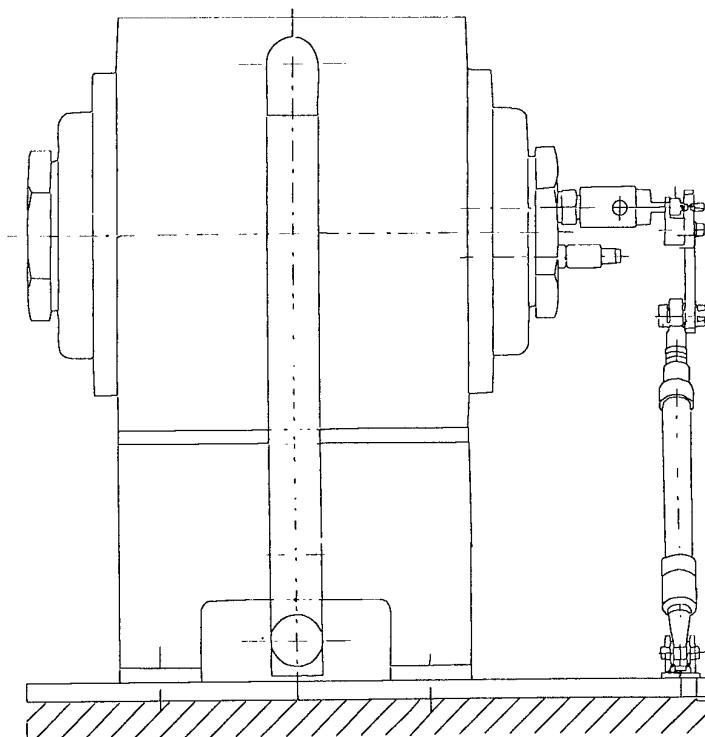


Figure A.7: Closed vessel.

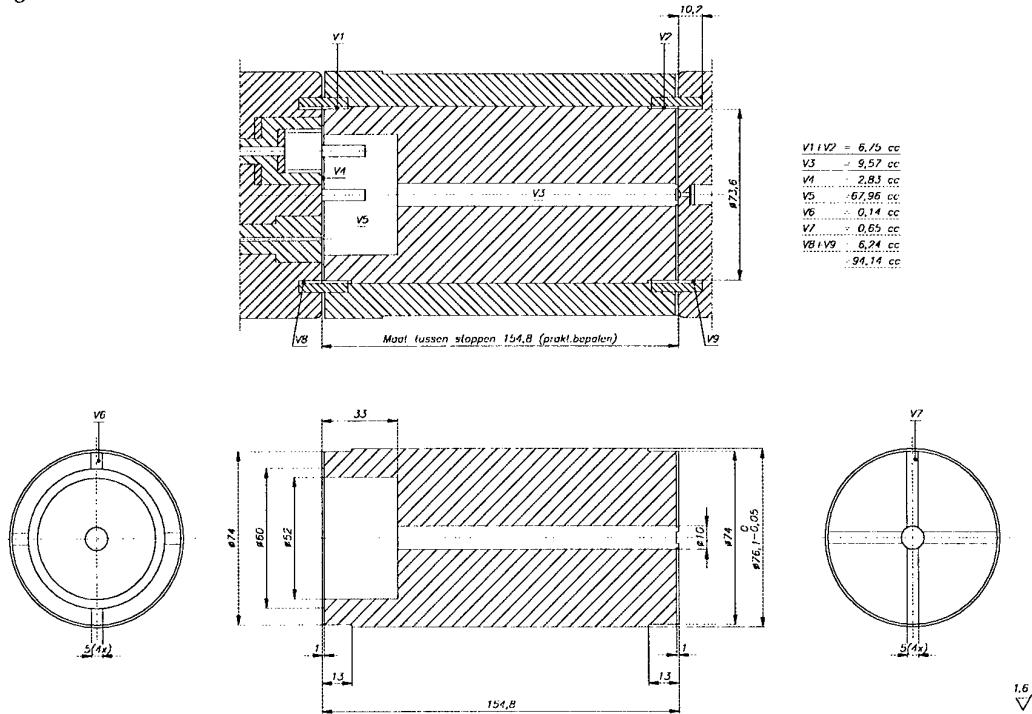


Figure A.8: Innerside of closed vessel.

Annex B Photos of the gas gun set-up

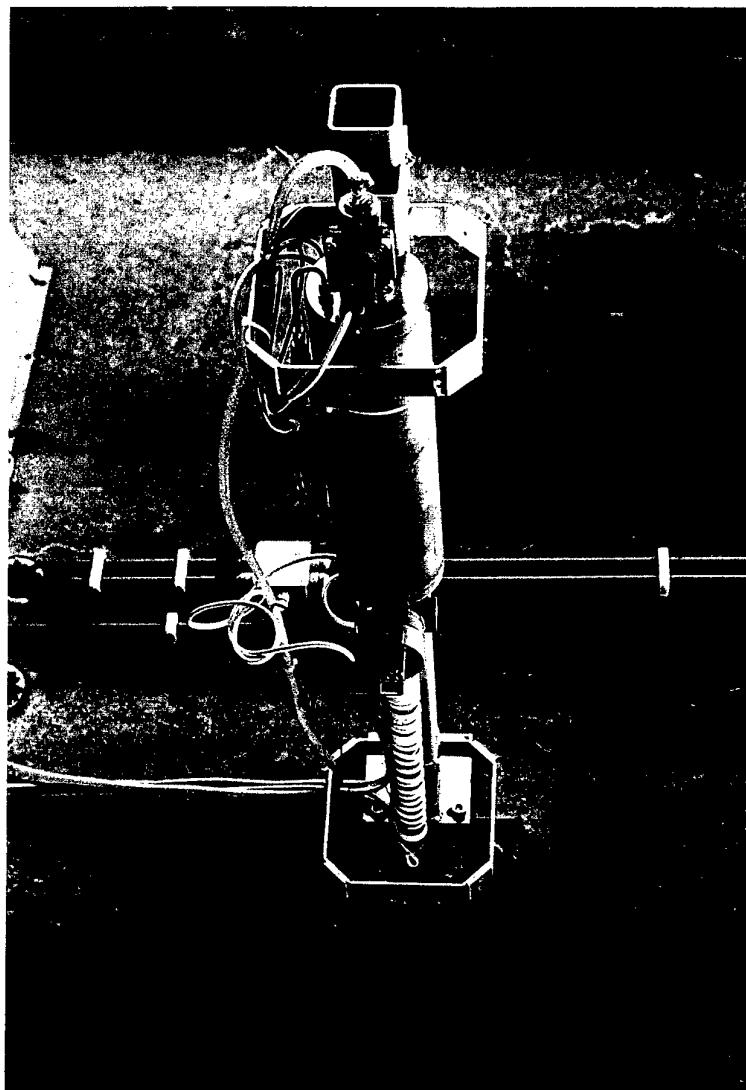


Photo B.1: A photo of the gasgun (a gas tank, a launching tube with two air valves) outside the test bunker.

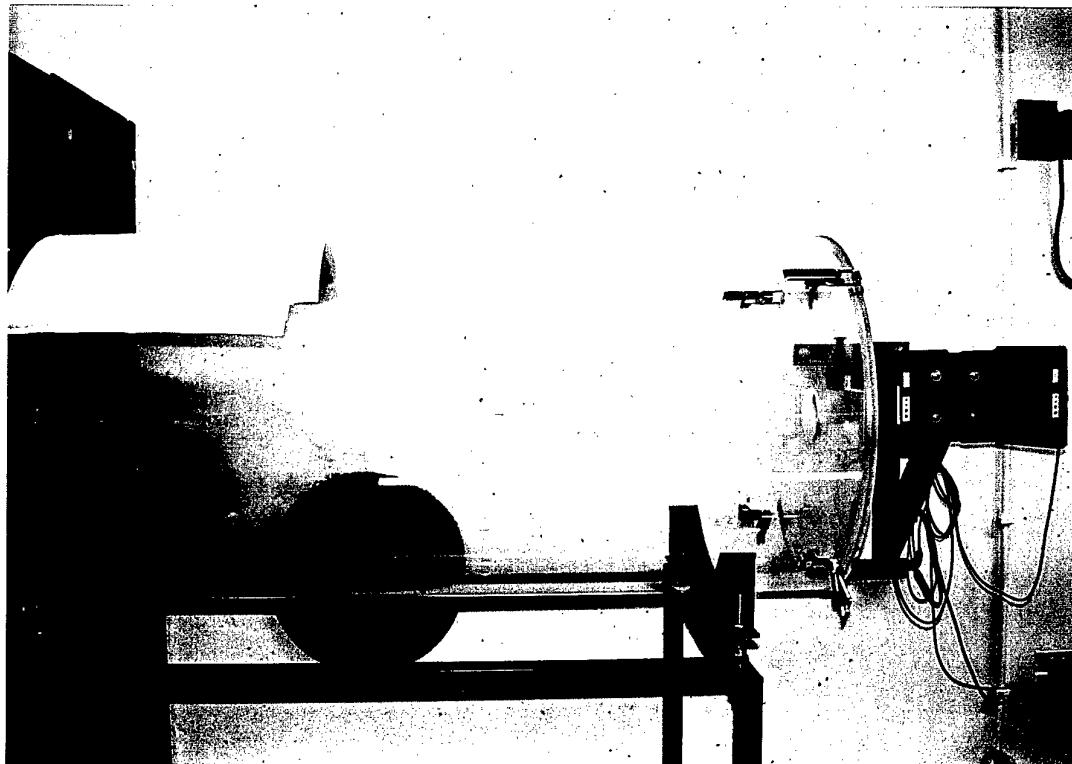


Photo B.2: The recovery box (middle), the optical barrier (right) and the impact plater (left) inside the test bunker.

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10. TITLE AND SUBTITLE
The construction of the friability test at TNO-PML

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14. SUPPLEMENTARY NOTES
The classification designation Ongerubriceerd is equivalent to Unclassified.

15. ABSTRACT (MAXIMUM 200 WORDS (1044 BYTE))
In co-operation with SNPE, France, the friability test has been constructed. This test is applied to determine the effect of impact on an explosive substance which is a candidate to be assigned as an Extremely Insensitive Detonating Substance (EIDS). In this test a sample is directed against a steel plate at a velocity of about 150 m/s by using a gas gun.
After having performed this test, the explosive is tested in the closed vessel test to assess any changes in the burning characteristic, i.e. the peak pressure and pressure rise rate, due to the impact.
The volume of the existing closed vessel needed to be adjusted in order to fulfil the calibration requirements with respect to peak pressure (80 ± 0.2 MPa) and pressure rise rate (not more than $15.2 \pm$ MPa/msec). This calibration was performed using a reference propellant delivered by SNPE. It appears that neither calibration requirement could be met by the closed vessel. The requirement for the peak pressure could be fulfilled, but not for the pressure rise rate.

16. DESCRIPTORS

Friability Test	Impact
Construction	Gas gun
Explosives	Combustion
Propellants	Closed vessel test
Sensitivity	Experimentation

17a.SECURITY CLASSIFICATION (OF REPORT) Ongerubriceerd	17b.SECURITY CLASSIFICATION (OF PAGE) Ongerubriceerd	17c.SECURITY CLASSIFICATION (OF ABSTRACT) Ongerubriceerd
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